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<b>13. SUPPLEMENTARY NOTES</b>						
<b>14. ABSTRACT</b> Continued improvements in membrane life and efficiency were realized through further optimization of the electrode attachment process. An MEA fabricated with 90 micron Gore membrane and Proton electrodes operating at 1.86 A/cm2 has reached 1000 hours of operation at 400 psi differential pressure and 80C with performance below 1.9V, meeting the program objectives. A 60 micron test continues to operate at similar conditions. All objectives have been met for the first phase of this project, with durability testing ongoing. Results are described in more detail in the following sections. New options on this project have been initiated and the next quarterly report will add the new tasks.						
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## **Quarterly Progress Report**

**Project Title:** Improved Round Trip Efficiency for Air Independent Regenerative Fuel Cell Systems

**Project Period:** June 18, 2010 to June 17, 2011

**Date of Report:** April 4th, 2011

**Recipient:** Proton Energy Systems

**Award Number:** N00014-10-C-0369

**Working Partners:** W. L. Gore

**Cost-Sharing Partners:** None

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**ONR Program Officer:** Maria Medeiros

**Project Objective:**

The purpose of this effort is to investigate advanced membrane materials that enable higher efficiency electrolysis, substantially improving the practical energy density for regenerative fuel cell applications. Additionally, exercisable options in this project will advance the understanding, implementation, and operational testing of the features that enable an RFC to simultaneously be truly air independent and have high energy density.

**Objectives:**

- Define the key membrane attributes that correlate with performance characteristics important for device function such as proton conductivity, ion exchange capacity, nitrogen and water permeation, and visual evaluation of mechanical strength of the membrane in the seal areas of the cell. (Gore and Proton)
- Determine the optimal processing parameters (pretreatment, pressing temperature, time) of these membranes for MEA fabrication. (Proton)
- Define thickness of the membrane required to withstand sealing loads and electrical loads as well as differential pressure. (Proton)
- Define the practical performance limits of these new membranes in terms of operating current, pressure, and temperature. (Proton)
- Based on screening of membrane samples, test a refined list of potential candidates at full-sized MEA scale.

**Background:**

Navy underwater vehicle platforms (UUV, ASDS, SWCS, etc.) are demanding larger and larger energy storage capacities to accommodate longer underwater missions and increased platform power requirements. New energy storage devices with high volumetric energy density for underwater vehicles, both manned and unmanned, are therefore needed, such as regenerative fuel cell (RFC) systems based on proton exchange membrane (PEM) technology. An RFC consists of a fuel cell powerplant, an electrolysis system for recharging the reactants, and reactant storage. These water-based energy storage systems have been shown to perform substantially better than traditional battery systems in areas such as rechargeability, specific energy density, and reliability. Advanced membrane and catalyst materials will enable higher efficiency electrolysis, substantially improving the practical energy density for regenerative fuel cell applications.

From a full proposal to develop an advanced demonstration system, Task 5 was selected for initial study. This task focused on membrane development. The research objectives for Phase 1 of this task were broken into the following separate subtasks:

**Task 5.0: Thinner, Reinforced Membranes:**

***Task 5.1 Alternative Membrane Material Procurement***

The contractor shall procure up to four advanced alternate membrane materials from not more than two membrane suppliers. These samples will be large enough for testing at the 0.03 ft<sup>2</sup> cell size.

***Task 5.2 Alternative Material Screening Tests***

The contractor shall evaluate the advanced alternate membrane materials for strength, fluid permeation, and ionic conductivity using typical Proton procedures. Fluid permeation may be conducted at up to two temperatures and three pressures.

***Task 5.3 Material Treatment Process Optimization***

The contractor shall conduct not more than three process trials with each of two alternative membrane candidates.

***Task 5.4 Catalyst Application Process Optimization***

The contractor shall conduct not more than three process trials with each of two alternative membrane candidates.

***Task 5.5 Feasibility Testing***

The contractor shall evaluate the samples generated from tasks 5.4 using standard Proton procedures for lateral and cross-cell resistance measurements. The best of the three trials for each candidate shall be selected for use in integrated operational testing. Not more than 3 single-cell tests shall be supported for up to 100 hours.

**Status:**

Continued improvements in membrane life and efficiency were realized through further optimization of the electrode attachment process. An MEA fabricated with 90 micron

Gore membrane and Proton electrodes operating at 1.86 A/cm<sup>2</sup> has reached 1000 hours of operation at 400 psi differential pressure and 80°C, with performance below 1.9V, meeting the program objectives. A 60 micron test continues to operate at similar conditions. All objectives have been met for the first phase of this project, with durability testing ongoing. Results are described in more detail in the following sections. New options on this project have been initiated and the next quarterly report will add the new tasks.

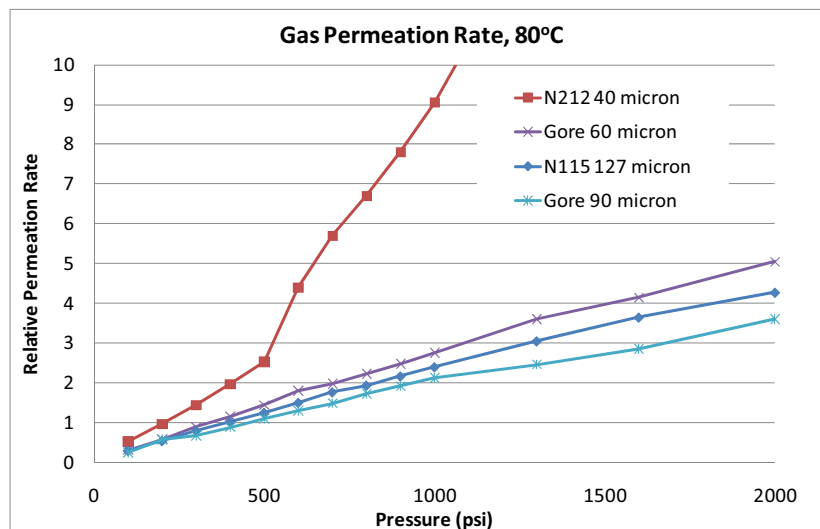
## **Task 5.0 Program Update: Thinner, Reinforced Membranes**

### ***Task 5.1 Alternative Membrane Material Procurement***

No new activity.

### **Subtask 5.2 Alternative Material Screening Tests**

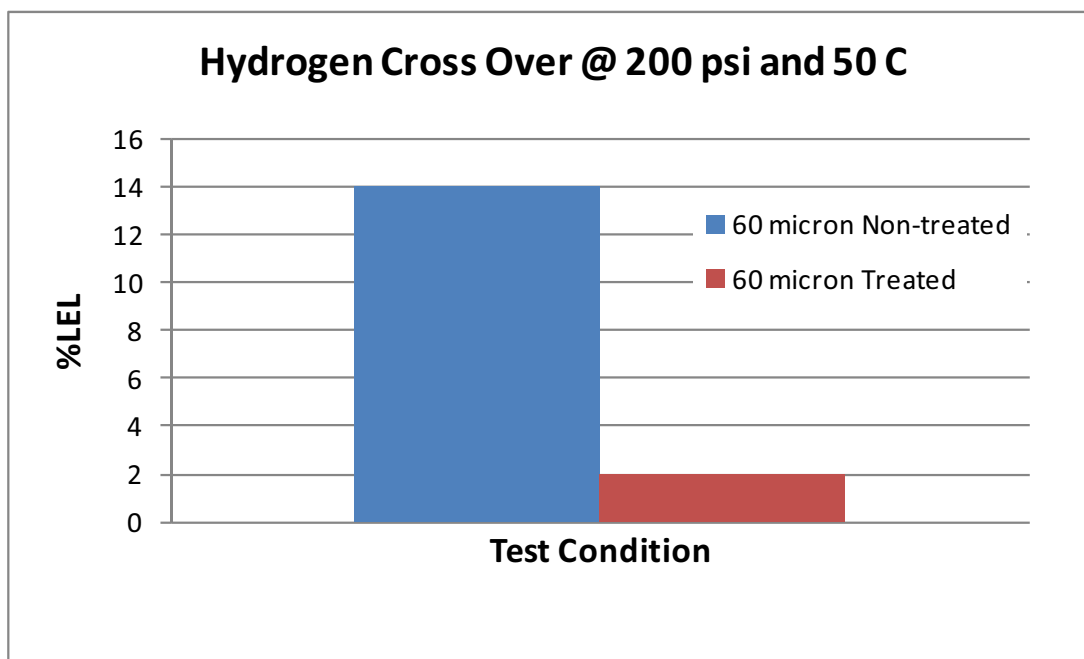
As previously reported, ionic conductivity for the WL Gore samples was very similar to the 5-mil Nafion sample. As an alternate comparison, results for 2-mil Nafion were plotted on the same scale, as shown below. The N212 sample was considerably higher in gas permeation than any of the other three samples, particularly at pressures above 500 psi. These results lend further confidence to the beneficial properties of the Gore material.



### **Subtask 5.3 Material Treatment Process Optimization**

Although Gore membranes exhibited gas permeation properties within the desirable range for unprocessed material, indicating good likelihood of success for integration into Proton's electrolysis cells, Proton currently uses a proprietary process on membranes to further reduce hydrogen crossover. Without this treatment, the amount of hydrogen that diffuses across the membrane typically becomes unacceptably high at low current densities such as idle mode operation. This process is also critical for enabling higher temperature and higher pressure operation of thinner membrane, where gas cross-over becomes a significant operational inhibitor. Studies were therefore performed to investigate application of this membrane treatment step to the Gore membrane prior to electrode attachment, to mitigate this hydrogen cross-over.

While there is still optimization of the process which needs to be performed, initial testing demonstrated feasibility of attaining typical crossover values. Proton continuously measures the amount of hydrogen gas in the oxygen stream as a safety check of stack and membrane integrity and utilized this measure as a comparison between treated and untreated samples. The graph below shows a comparison for the Gore 60 micron membrane.



#### **Subtask 5.4 Catalyst Application Process Optimization**

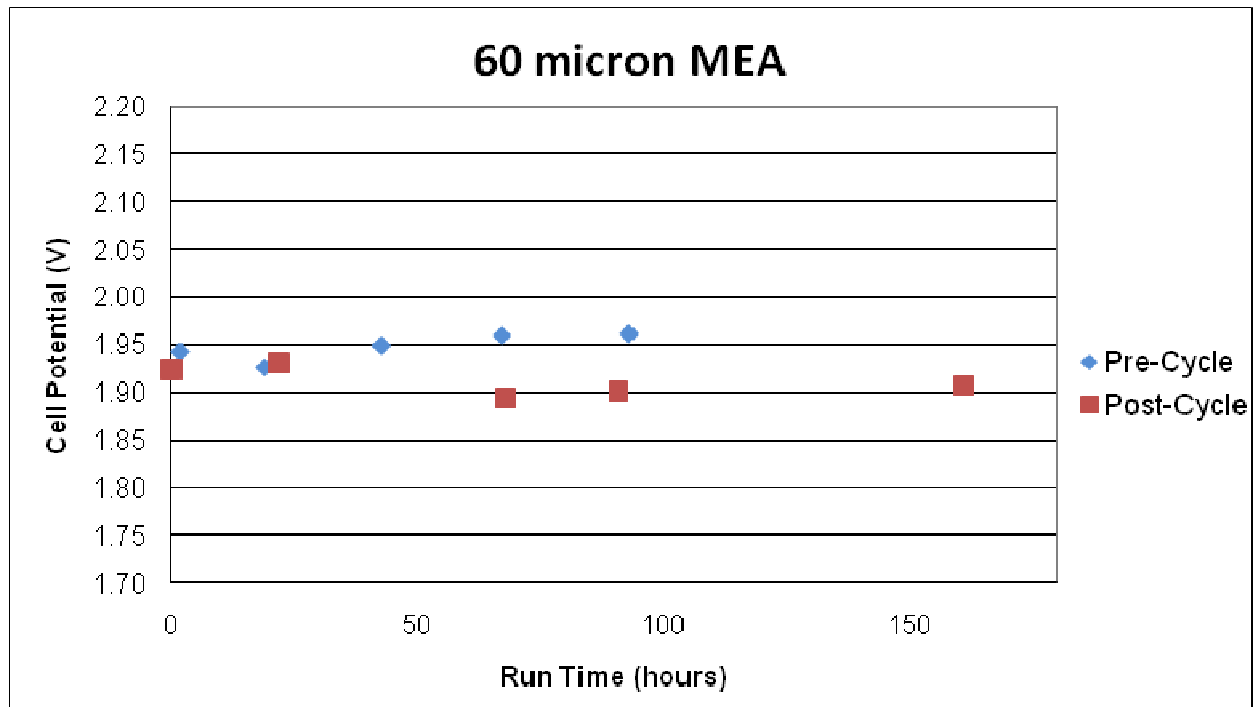
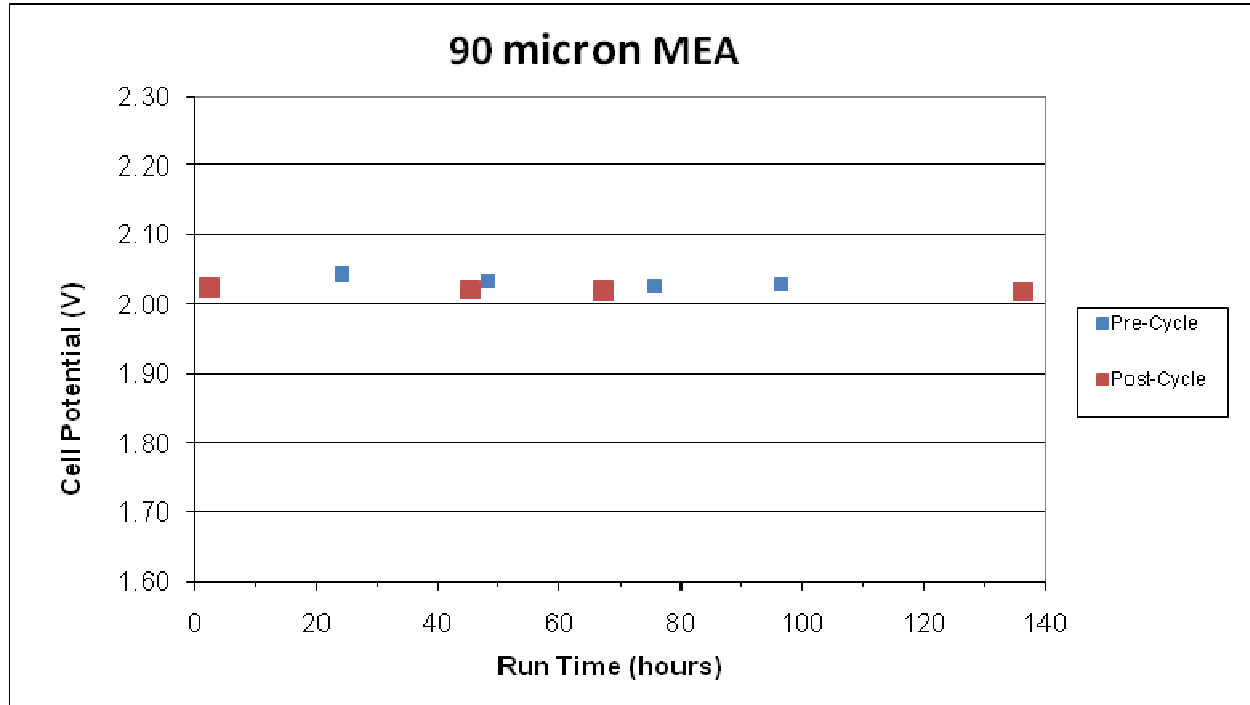
As described in the last report, an alternate pressing process with Proton's patented ink formulation resulted in the successful attachment of electrodes, without reducing membrane integrity. A set of these alternate press load conditions were used in the processing of full MEAs on 45, 60 and 90 micron Gore samples. The resulting MEAs passed substrate transfer and electrode adhesion tests prior to being assembled in a Proton cell stack based on our commercial production lab line architecture.

#### **Subtask 5.5 Feasibility Testing**

The MEAs described above were subjected to Proton's accelerated stress test protocol. Cycle testing was conducted at the steady-state conditions of 50 C and  $1.86 \text{ A/cm}^2$ , while operating pressure was cycled from zero to 400 psi, 300 times. While the 45 micron samples failed for high hydrogen cross-over during the cycle testing, the 60 and 90 micron samples successfully passed this stage. After the cycling protocol was completed, the cell stacks were assessed per Proton internal acceptance testing, which is used in the verification of all commercial product, to check for any signs of premature failure. None was identified in either the 60 or the 90 micron test.

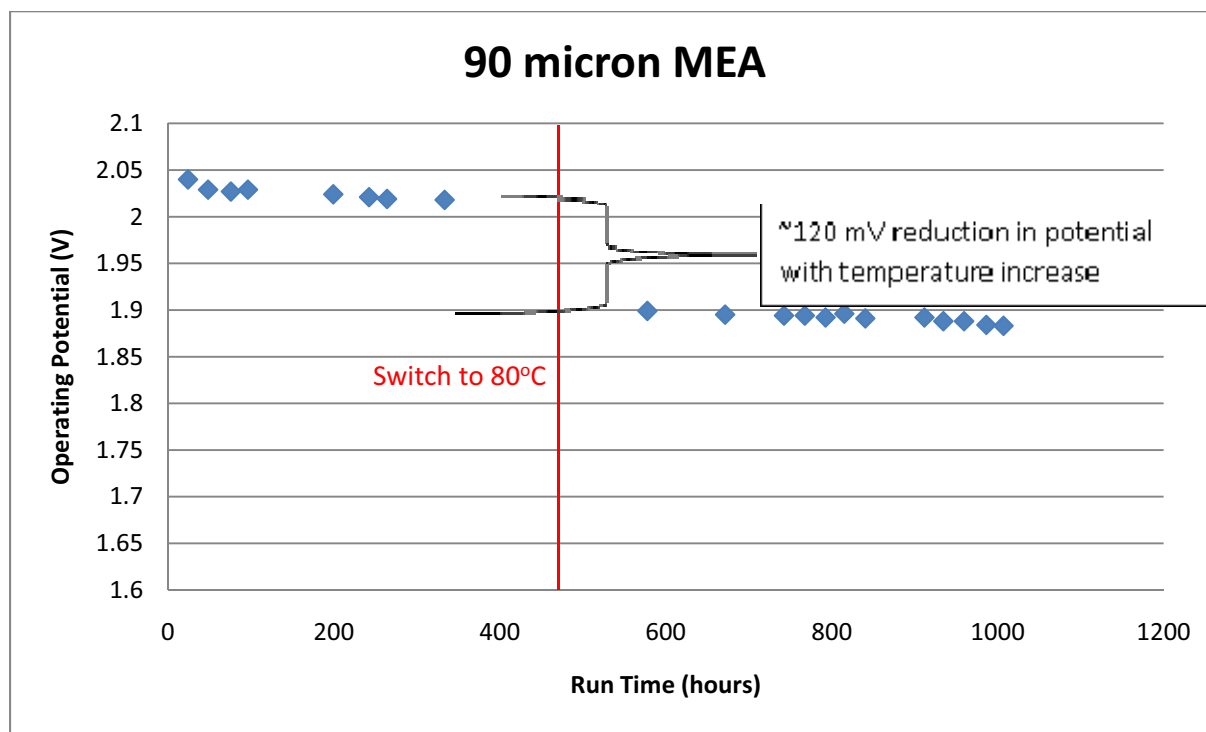
Another measure made during the stack operation to support the acceptance testing was the comparison of pre- and post operational voltage trends to look for degradation of performance, as a result of the cycle testing. Prior to the start of each cycle test, the

stacks were operated for 100 hours at 1.86 A/cm<sup>2</sup>, 50 C, and 200 psi to baseline the stacks. After cycle testing, the stacks were run for another 100 hours under the same conditions and the plots were compared. Each of the two graphs for the 60 and 90 micron test are shown below.



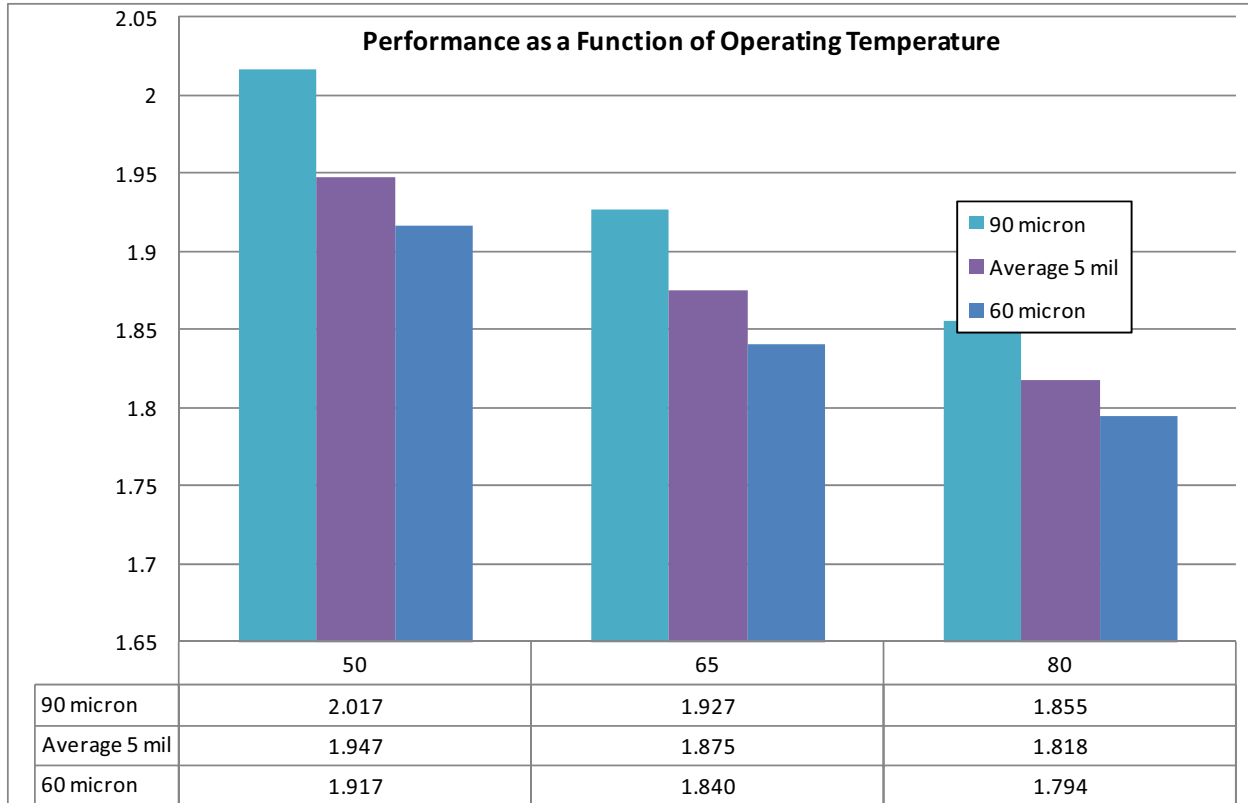
As shown in the graphs, there was no increase in potential observed in the post-cycle operation, indicating no performance penalty incurred at this point in the MEA life. This is an indication of the robustness of both the membrane and the electrode attachment process.

The samples were allowed to continue operation after the accelerated testing was completed. Pressure and temperature were increased to 400 psi and 80°C during the steady-state testing and the stack was operated under these conditions until 1000 hours of time had accumulated on the stack. The graph for the 90 micron sample is shown below.



The 60 micron test is still in the process of completing its 1000 hour test and will be included in the final report.

Operating potentials for each the 60 and 90 micron stacks were collected to characterize their behavior at various temperatures. The results from these tests were graphed against 5-mil Nafion, which has better performance than Proton's current commercial stack but does not pass the accelerated stress testing protocol discussed above. The comparison graph is shown below.



The 60 micron sample outperformed the 90 micron and 5-mil Nafion, so successful completion of the 1000 hour test will be critical in validating this optimal combination of Gore 60 micron and Proton electrodes. Testing continues at the time of this update.

#### **Task 5.0 Project Management and Reporting**

The Principal Investigator attended the ONR Program Review in March. The final report for the tasks detailed in this update is being drafted. Gore visited Proton's facility in February to discuss testing results.

#### **Plans for Next Quarter and Key Issues:**

In the next quarter, 1000 hours testing will be completed on the 60 micron sample and the final report will be completed. The next phase of work will focus on demonstration of the closed loop regenerative fuel cell system at Proton, beginning with refurbishment of the unit.

**Patents:** None to date.

#### **Publications / Presentations:**

No new presentations during the reporting period.



## Task Schedule

Task Number	Project Milestones	Task Completion Date				Progress Notes
		Original Planned	Revised Planned	Actual	Percent Complete	
1	Alternative Membrane Procurement	08/31/10	10/29/10	10/29/10	100%	Completed
2	Alternative Membrane Screening	09/30/10	2/15/11		100%	Diffusion and thickness measurements completed.
3	Membrane Treatment Optimization	12/31/10	12/21/10		100%	Range of press temperatures and durations determined
4	Catalyst Application Optimization	02/28/11	2/28/2011		100%	Inked electrodes show improved performance and durability
5	Feasibility Testing	06/17/11	04/25/11		95%	Have demonstrated 1000 hours on 90 micron and waiting for 60 micron test to finish
6	Project Management	06/17/11	04/26/11		90%	Complete and submit final report

**Budget Summary: will be updated with additional \$200K exercised in next report**

Quarter	From	To	Estimated Billing	Actual Billing
1Q10	06/18/10	9/30/10	\$30,190	\$34,992
2Q10	10/01/10	12/31/10	\$17,500	\$25,723
3Q10	1/1/11	3/31/11	\$15,824	\$15,065
4Q10	4/1/11	6/30/11	\$1158	
		<b>Totals=</b>	<b>\$76,938</b>	<b>\$75,780</b>